Laboratory Experiment Proposal Submission

Experimental Details

Experiment location: E138
Experiment title: Jeff Testing Kimani's Experiment - If Kim Sees This, It Was Successful
Experiment date: June 21, 2012
Experiment contactname: Kimani A. Stancil
Experiment contactphone: (301) 975-6650
Experiment contactemail: jkrzywon@nist.gov

Chemicals Used

Chemical Name	Health	Flammability	Reactivity	Special Hazards
N-isopropylacrylamide	Blank	Blank	Blank	NONE
Methylene Bisacrylamide	Blank	Blank	Blank	
2,2â€™ â€“Azobisisobutronitrile	Blank	Blank	Blank	NONE
Dioxane	Blank	Blank	Blank	NONE
Methacrylic Acid	Blank	Blank	Blank	NONE
Lead Methacrylate	Blank	Blank	Blank	NONE

Reactants and Resulting Samples

Chemical Name	Hazardous?	Known Hazards
Poly N-isopropylacrylamide Block Poly Methylene Bisacrylamide	N	

Required Safety Equipment

- [x] Glove Hot
- [x] Glove Neoprene

Required Laboratory Equipment

- [x] Hot Plate
- [x] Temperature Bath
- [x] Ultrasonic Bath

Experimental Write Up

Goal: To prepare 3-hydrogel types: 1) NIPAm-BIS, 2) NIPAm â€“BIS â€“ MAAc, or 3) NIPAm-BIS-Pb(MAAc)2 using dioxane for synthesis and AIBN as initiator. The target aim is to prepare hydrogel in flat planar shape for efficient use in neutron scattering experiments.

Recipe
Major Monomer(s): 6M = 6000 mM NIPA(N-isopropylacrylamide),
[20, 60, 100, 150, 200] mM BIS (methylene bisacrylamide)
Initiator: 10 uM AIBN (2,2â€™ â€“Azobisisobutronitrile)
Solvent (Synthesis Medium): Dioxane (20% by Volume = 4 ml per batch)
Minor Monomer(s): Gel type 2) {12, 25, 50, 100} mM MAAc (methacrylic acid)
Gel type 3) {6, 12.5, 25, 50} mM Pb(MAAc)2 (lead methacrylate)
Other chemicals for preparation and characterization:
4-(2-Pyridylazo)resorcinol will be used for small scale detection of Pb amounts in washing procedure, step 11) below. Also in Step 11), hydrochloric acid (HCL), sodium hydroxide (NaOH), and water (H2O), and deuterated water (D2O) will be used.
Materials: Thermal heated water baths. Ultra sound and heat baths. Hot plate and crystallization dish. Spatulas, test tubes, 20 ml glass vials, glass pippetes for transfer. Freezer.

Preparation: Mix majority monomers. Prepare minority monomers. Add initiator, then polymerize.
Prepare gel for experimental use.
Before or in parallel with steps 1) â€“ 4) below, prepare 10 uM AIBN initiator = 0.1631g AIBN in 1 ml Dioxane.
1) Use a 50 ml graduated cylinder to mix powdered NIPAm and BIS.
Example: 20 mM BIS = 0.0617 g BIS + 13.4878 g NIPAm
2) Add 4 ml dioxane. Cover graduated cylinder with parafilm and aluminum foil. Place in sonicated heat bath water at 60o C for 2 hours.
3) In 20 ml glass vials, place either liquid MAAc (i.e. gel type 2)) or powder Pb(MAAc)2 (i.e. gel type 3). Example: 6mM Pb(MAAc)2 = 0.0240 g, 12 mM MAAc = 0.01105 g. Note that for gel-type 1 (NIPAm â€“ BIS), an empty vial is used.
4) When solution in step 2) is ready, it is divided equally by volume into vials containing only MAAc or Pb(MAAc)2 . In the case of gel-type 1, the whole solution can be distributed evenly into test tubes (step 5) below) or glass vials. Then place vials in sonicator and heat bath water at 55o C. (1 hour)
5) Transfer solution to 8 ml test tubes or pre-gel molds. Seal test tubes (with rubber stops) or molds with suitable covers. Tubes and molds should be reintroduced to heat, i.e. water bath, or hot plate (55o C) depending on gel mold. Add 80 ul of 10 uM AIBN solution to each test tube.
6) Immediately place test tubes in freezer to retard polymerization and to stimulate nitrogen removal. Prepare heat bath without sonication at 60o C.
7) After 1-hour, place test tubes under vacuum for 10-15 seconds to remove nitrogen.
8) Immerse sealed test tubes heat bath without sonication at 60o C. The estimated polymerization time is 12-14 hours with 10-12 hour curing for a 1 day duration in heat bath.
9) Using fume hood, remove gels from test tube molds by hammer to crack tubes gently.
10) Slice test tube gel into thin disks for study. Immerse all disks in H2O for 3 days with solution changes every half day (minimum).
11) Disk Treatment 1 = vacuum dry 3-4 days, then place in cells and add D2O for study. Disk treatment 2 = 100 mM HCl (soak 2 days) ïƒ 100 mM NaOH (soak 2 days) ïƒ H2O soak 3 days ïƒ vacuum dry ïƒ Immerse in D2O . All soaking durations are maximum estimates based on 0.5 mm diameter cylindrical gel pieces where diffusion may be more limited.
12) For flat gel from mold, repeat step 5) above by the following:
a) Lay wafer mold on hot plate.
b) Deposit solution on in mold.

c) Add initiator by using a volume, V, less than 80 ul used for the test tube mold. Basic estimate is V= (mold volume(ml))/100 .

d) Lid flat mold with Aluminum (Al) foil and freeze for 1 hour.

e) Remove from freezer, and lightly punch holes in foil. Vacuum gently over holes.

f) Place wafer mold and pre-gel on hot plate. Cover with crystallizing dish, and set temperature to â‰¥ 60o C. Monitor process (1st time to decide on duration; default will be 24 hours).

g) Repeat step 11) above for final preparation.

Experimenter Signature: _____ Date: _____

Lab Responsible Signature: _____ Date: _____